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Indian Standard SPECIFICATION FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

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INDIAN STANDARDS INSTITUTION
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Medice

Continued from

Indian Standard

SPECIFICATION FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

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Indian Standard

SPECIFICATION FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

O. FOREWORD

- **0.1** This Indian Standard was adopted by the Indian Standards Institution on 30 November 1985, after the draft finalized by the Ceramic ware Sectional Committee had been approved by the Chemical Division Council.
- **0.2** Silicon carbide is the hardest commercially available substance, its hardness being between 9.0 to 9.6 on Moh's scale and is next to diamond. Silicon carbide (SiC) is manufactured by the reaction of sand and coke in a resistance type electric furnace.
- **0.3** There are two types of silicon carbide manufactured commercially, known as green variety and black variety. The green variety finds its major uses in lapping, polishing, grinding, processing of tungsten carbide, and glass, etc, and the black variety in the bonded, abrasive metallurgical refractory applications, and manufacture of crucibles, etc.
- **0.4** At present, there exists no uniform practice for designating the grit size of silicon carbide. Silicon carbide grains are classified by reference to both grade and grit numbers and this practice has been found to be somewhat confusing. This standard recognizes only grit numbers as the basis for designating grain sizes. Grit numbers 8 to 220 are popularly known as grains and those finer than and excluding grit number 220 are known as micro grits.
- **0.5** In formulation of this standard considerable assistance has been derived from the Specification of Federation of European Producers of Abrasives (FEPA).
- **0.6** For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

^{*}Rules for rounding off numerical values (revised).

18:11643-1985

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for black and green varieties of silicon carbide of grit number 8 to 220 for bonded abrasive products.

2. TERMINOLOGY

- 2.1 For the purpose of this standard, the following definitions shall apply.
- 2.1.1 Grit Number Indicates the size of silicon carbide abrasive grain.
- 2.1.2 Grain Size Distribution The percentage of grits of different sizes of silicon carbide abrasive grades composing the powders.

3. TYPES

3.1 Silicon carbide shall be of the following two types:

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Type 1 — Black silicon carbide (SiC — Black)
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Type 2 — Green silicon carbide (SiC — Green)

4. REQUIREMENTS

- **4.1 Description** The material shall be in the form of powder, free from contamination like clay material, pebbles and other extraneous matters.
- **4.2** The material shall be of grit number 8 to 220 in step as indicated in Table 1.
- **4.3 Grain Size** The material shall conform to the grain size distribution as given in Table 1 for a particular grit number when tested as per the method prescribed in Appendix A.
- **4.4** The material shall comply with the physical requirements given in Table 2 when tested in accordance with the methods prescribed in Appendix B.
- **4.5** The material shall also comply with the chemical requirements given in Table 3 when tested in accordance with the methods prescribed in Appendix C.

5. PACKING AND MARKING

5.1 Packing — The material of one type and size of grit number shall be packed in containers adequately protected against contamination during transit as agreed to between the purchaser and the supplier.

TABLE 1 GRAIN SIZE REQUIREMENT FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

(Clauses 4.2 and 4.3)

SL GRIT No. No.		FIRST SIEVE		SECOND SIEVE		Tı	THIRD SIEVE		Тн		ND FOURTH	FIFTH SIEVE					
NO.	No.	Sieve	e 1 (Oversize	Siev	e 2	Oversize	Sieve 3		Oversize	Sieve 4 Cumulative Oversize on Sieves 3 + 4					Undersize	
		mm	μm	percent, Max	mm	μm	percent, Min	mm	μm	percent, Min	mm	μm	percent, Min	mm	μm	percent, <i>Max</i>	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	
i)	8	4.00		0	2.80	_	15	2.36	_	45	2.00		80	1.70		3	
ii)	10	3·3 5	_	0	2.36		15	2.00		4 5	1.70		80	1.40	_	3	
iii)	12	2.80	_	0	2.00		15	1.70	_	45	1.40	_	80	1.18		3	
iv)	14	2·3 6	_	0	1.70		15	1.40		45	1.18		80	1.00		3	
v)	16	2.00	_	0	1.40	_	15	1.18	_	45	1.00	_	80	. —	850	3	
vi)	20	1.70		0	1.18	_	15	1.00		45	_	850	80	_	710	3	
vii)	24	1.18		0	_	850	25	-	710	45	_	600	70		500	3	
viii)	30	1.00		0	_	710	25	_	600	45	_	500	70		4 25	3	
ix)	3 6	_	850	0	_	600	2 5	_	500	45	_	425	70	_	355	3	
x)	46		600	0		425	30		355	40		3 00	65		250	3	
xi)	54	—	500	0	—	355	3 0	—	3 00	4 0	-	25 0	65		212		
xii)	60	_	425	0	– ′	300	30		250	40		212	6 5	-	180	_	
xiii)	70	_	355	0	_	250	25		212	40	_	180	65		150	3	
xiv)	80		300	0	_	212	25	_	180	40	-	150	65		125	3	
xv)	90	_	250	0		180	20	_	150	40	_	125	65		106	3	
xvi)	100		212	0		150	20	—	125	4 0	_	106	65		75	3	
xvii)	120		180	0	_	125	20		106	40	_	90	65		6 3	3	
															((Continued)	

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TABLE 1 GRAIN SIZE REQUIREMENT FOR SILICONE CARBIDE FOR BONDED ABRASIVE PRODUCTS — Contd

IIIIIII I I KODOGIS — Ooma															
SL No.	Grit No.	Sieve		SIEVE Oversize			Sieve 	Тн	SIE	D FOURTH	F)FTH	OURTH AND SIEVES		H SIEVE
								Sieve 3	Sieve 4	Cumulative Oversize on Sieves 3+4		ze 5 (Cumulative Oversize on Sieves 3+4+5		size
		mm	μm	percent, Max	mm	μm	percent, Min	mm	$\mu \mathbf{m}$	percent, <i>Min</i>	mm	μm	percent, Min	μm	percent, Max
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)	(15)	(16)
xviii)	150	_	150	0	_	106	15	90	75	4 0	_	63	65	45	3
xix)	180		125	0	_	90	15	75	6 3	40	_	53	65		_
xx)	220	_	106	0		75	15	6 3	53	40	_	4 5	60		_

TABLE 2 PHYSICAL REQUIREMENTS FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS (Clause 4.4)

SL No.	Grit No.	Loose Pack: g/i			etic Iron, ent, <i>Max</i>	Transmittance Percent, <i>Min</i>		
		SiC Black	SiC Green	SiC Black	SiC Green	SiC Black	SiC Green	
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	
i)	8	*	*	0.03	0.03	60	60	
ii)	10	*	*	0.03	0.03	60	60	
iii)	12	*	*	0.03	0.03	60	60	
iv)	14	1.48-1.56	*	0.03	0.03	60	60	
v)	16	1.50-1.58	*	0.03	0.03	60	60	
vi)	20	1.52-1.60	*	0.03	0.03	60	60	
vii)	2 4	1.52-1.60	*	0.03	0.03	60	60	
viii)	30	1.52-1.60	*	0.03	0.03	45	45	
ix)	3 6	1.52-1.60	1.50-1.58	0.03	0.03	45	45	
x)	46	1.52-1.60	1.50-1.58	0.03	0.03	45	45	
xi)	54	1.52-1.60	1.52-1.60	0.03	0.03	30	30	
xii)	60	1.52-1.60	1.52-1.60	0.03	0.03	30	30	
xiii)	70	1.50-1.58	1.50-1.58	0.03	0.03	30	30	
xiv)	80	1.48-1.56	1.50-1.58	0.03	0.02	30	3 0	
xv)	90	1:46-1:54	1.48-1.56	0.03	0.02	15	15	
xvi)	100	1:46-1:54	1.48-1.56	0.03	0.02	15	15	
xvii)	120	1.47-1.55	1.47-1.55	0.03	0.02	15	15	
xviii)	150	1.45-1.53	1.43-1.51	0.02	0.02	15	15	
xix)	180	1.43-1.51	1.41-1.49	0.02	0.02	10	10	
xx)	220	1.41-1.49	1.40-1.48	0.02	0.02	10	10	

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TABLE 3 CHEMICAL REQUIREMENTS FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

(Clause 4.5)

SL No.	Requirement	SiC-Black	SiC-Green	METHOD OF TEST (REF TO CL NO. IN APPENDIX)
(1)	(2)	(3)	(4)	(5)
i)	Free silicon (Si), percent by mass, Max	0.5	0.2	C-1
ii)	Free carbon (C), percent by mass, Max	0.4	0.5	C-2
iii)	Free SiO ₂ , percent by mass, Max	1.0	0.5	C-3
iv)	Silicon carbide (SiC), percent by mass, Min	97:5	98·5	C-4
v)	Surface iron oxide (Fe ₂ O ₃), percent by mass, Max	0.1	0.1	C -5
vi)	Aluminium oxide (Al ₂ O ₃), percent by mass, Max	0.5	0.1	C -6

5.2 Marking — The container shall be marked with the following:

- a) Name of the material;
- b) Type;
- c) Grit Number;
- d) Mass of the material;
- e) Manufacturer's name or his recognized trade-mark, if any;
- f) Batch No. or a suitable code No. to enable the lot to be traced back from records; and
- g) Month and year of manufacture.

5.2.1 The containers may also be marked with the ISI Certification Mark.

Note — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

- 6.1 Representative samples shall be drawn as prescribed in Appendix D.
- **6.2 Test Sample** Grind 10 to 20 g of the representative sample in a steel mortal so that it shall pass through 150 micron mesh and use it for the testing of chemical analysis.

7. TEST METHOD

- 7.1 Tests shall be conducted as prescribed in Appendix A, B and C.
- 7.2 Quality of Reagents Unless specified otherwise, pure chemicals and distilled water (see IS: 1070-1977*) shall be employed in the test.

Note: "Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(Clause 4.3 and Table 1)

DETERMINATION OF GRAIN SIZE

A-0. OUTLINE OF THE METHOD

A-0.1 The material is sieved through a nest of standard test sieves and the percent of material passing through various sieves calculated.

A-1. EQUIPMENT

- A-1.1 Test Sieves See IS; 460 (Part 1)-1985†. Aperture sizes to be used as indicated in Table 1.
- A-1.2 Mechanical Shaker A suitable mechanical shaker, rotap type, electrically operated with a speed of approximately 250 revolution per minute and about 150 taps per minute, attached with a timer may be used. The timer may be able to operate at least for ten minutes having an accuracy of ± 5 second.
- A-1.3 Balance The balance shall have an accuracy of ± 0.1 g.

A-2. PROCEDURE

A-2.1 Clean and thoroughly dry the test sieves before use. The desired nest of sieves used for testing each grit, category (see Table 1) is assembled in order of mesh size with the coarsest sieve on the top, progressing to the finest with a receiver on the bottom and lid at the top.

^{*}Specification for water for general laboratory use (second revision).

A-2.2 Weigh accurately about 100 g of the well mixed sample and transfer it to the top sieve. The entire unit placed in the mechanical shaker with a lid and a receiver. The timer is set for 10 minutes and turned on. At the completion of the cycle, the nest of the sieves are removed from the mechanical shaker. Beginning with the top (coarset) sieve, take out the portion of the grain retained on to a clean petri dishes with the help of camel hair brush separately, weigh the material collected from each sieve. Calculate the percent of material retained on each sieve.

A-3. CALCULATION

A-3.1 Material retained on any given given percent by masss = Mass in g of the material retained on the given sieve retained on the given sieve × 100 Mass of the material taken for the test

APPENDIX B

(Clause 4.4 and Table 2)

TEST METHODS FOR PHYSICAL REQUIREMENTS OF SILICON CARBIDE FOR BONDED AND COATED ABRASIVE PRODUCTS

B-1. DETERMINATION OF LOOSE PACKED DENSITY

B-1.0 Outline of the Method — A material is allowed to fall free in a cylinder of known volume and the mass of the given volume is obtained. The result is expressed in grams per centimetre.

B-1.1 Apparatus

- **B-1.1.1** Loose Packed Density Apparatus as illustrated in Fig. 1.
- **B-1.1.2** Cylinder of Brass 150 ml.
- **B-1.1.3** Beaker 150 ml (see IS: 2619-1971*).
- B-1.2 Calibration of Brass Cylinder Prior to use, determine the volume of the brass cylinder as follows:

'Weigh the dried empty cylinder with a flat glass plate slightly larger than the diameter of the cylinder. Fill the cylinder with distilled water and slide the glass plate in contact with the upper edge of the cylinder, in such a way to remove excess water. Held the

^{*}Specification for glass beakers (first revision).

glass plate firmly inplace, wipe off the excess water and obtain the gross mass. The volume of the cylinder is calculated using the following formula:

$$V = \frac{M}{\text{Density of water at test temperature}}$$

where

V = volume of cylinder in ml, and

M = net mass of water in g.

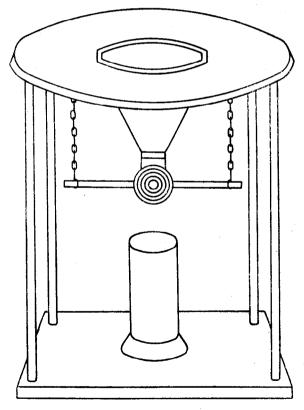


Fig. 1 Loose Packed Density Apparatus

B-1.3 Procedure — Place the sample of silicon carbide grain to be tested in the 150 ml beaker, level fulf. With the funnel outlet closed by the rubber ball, placed properly centered cylinder under the outlet of the funnel on felt or rubber pad, firmly pour the sample from the beaker into the funnel. Trip the rubber ball to the open position and allow the grain to fall freely to fill the cylinder. Immediately after the funnel is emptied, smoothly slide the glass plate across in contact with the top edge of the cylinder to level the surface of the grain. Care must be taken to avoid vibration, shock or any other disturbances during the test. Carefully brush the grain off the cylinder and its base. Weigh the cylinder with the sample. Repeat at least three times this test and report the average of the results.

B-1.4 Calculation

Calculate the loose packed density LPD as follows:

Loose packed density, g/ml = $\frac{M_1}{V}$

where

V = volume of cylinder in ml, and

 M_1 = net mass of the grain content in the cylinder in g.

B-2. DETERMINATION OF MAGNETIC IRON CONTENT

B-2.1 Apparatus — A magnetic analyzer.

B-2.2 Procedure — A representative sample of the material is taken in the test container and placed in the cavity provided in the analyzer. The reading displayed on the digital display taken as the percent magnetic iron value. The equipment is calibrated from time to time using the standard sample.

B-3. DETERMINATION OF TRANSMITTANCE

B-3.0 Principle of the Method — Transmittance of light through water after in contact with the material is compared with transmittance of light through distilled water which is taken as hundred percent.

B-3.1 Apparatus

- B-3.1.1 Photocolorimeter
- B-3.1.2 Mechanical Shaking Machine
- B-3.2 Procedure Take 7 g of the sample in a 100 ml volumetric flask and add 100 ml of distilled water. Shake it for 5 minutes and decant the water from the top into a test cell. Measure the percentage light transmitted using blue or green filter. Compare the light transmitted through distilled water taken as hundred percent.

APPENDIX C

(Clause 4.5 and Table 3)

TEST METHODS FOR CHEMICAL REQUIREMENTS OF SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

C-1. DETERMINATION OF FREE SILICON

C-1.1 Reagents

- **C-1.1.1** Ferric Ammonium Sulphate Indicator Solution Dissolve 20 g of ferric ammonium sulphate [NH₄Fe (SO₄)₂.2H₂O] in water containing 5 ml of nitric acid and dilute to 100 ml.
- **C-1.1.2** Potassium Thiocyanate (0.1 N) Dissolve 9.712 g of potassium thiocyanate (KCNS) in water. Transfer the solution in a 1 000 ml volumetric flask and dilute to volume with water. Standardize the solution by titrating against standard silver nitrate solution using the ferric ammonium sulphate as indicator.
- **C-1.1.3** Silver Fluoride Solution Dissolve 10 g silver oxide (Ag_2O) in about 500 ml of hydrofluoric acid (48 percent v/v). Allow the solution to stand for at least 24 h. Filter the solution before use.
 - **C-1.1.4** Diluted Hydrochloric Acid -1:3 (v/v) (see IS: 266-1977*).
 - **C-1.1.5** Diluted Nitric Acid 1 : 3 (v/v) (see IS : 264-1976†).
- **C-1.2 Procedure** Weigh about 2 g of the test sample into a 150 ml beaker and add 60 ml (see 6.2) of dilute hydrochloric acid. Boil the solution for ten to fifteen minutes. Filter the solution through a filter paper using polyethylene funnel having a 70 mm stem to which a flexible plastic tubing of 100 mm length is fastened. Wash the paper with water until the filter is free from chlorides.
- C-1.2.1 After the solution in the filtrate funnel has completely drained attach the pinch clamp to the tube fastened to the funnel. Fill the filter paper with the silver fluoride solution (C-1.1.3). Allow the solution to remain in the funnel for at least one hour.
- C-1.2.2 Drain the solution into a beaker. Wash the filter paper with water until the filtrate is free of silver salts. Discard the filtrate and washings.
- C-1.2.3 Dissolve the metallic silver in the filter paper by pouring 100 ml of hot dilute nitric acid (C-1.1.5) through the paper. Collect the resulting solution in a 250 ml conical flask. Wash the filter paper

^{*}Specification for hydrochloric acid (second revision).

[†]Specification for nitric acid (second revision).

with water. Dilute the solution to 150 ml. Add 1 ml of the ferric ammonium sulphate indicator solution (C-1.1.1). Titrate the solution to the first persistent pink colour with standard potassium thiocyanate (C-1.1.2).

C-1.3 Calculation

Free silicon, percent by mass = $\frac{V \times 0.7022 \times N}{M}$

where

V = volume of standard potassium thiocyanate solution required for titration,

N =normality of standard potassium thiocyanate solution,

M =mass in g of the sample.

C-2. DETERMINATION OF FREE CARBON

C-2.1 Apparatus

C-2.1.1 High Temperature Ceramic Combustion Boat — (See IS: 3953-1966*).

C-2.1.2 Tube Furnace

C-2.1.3 Absorption Assembly

C-2.2 Procedure

C-2.2.1 Purge the absorption assembly with oxygen for 10 to 15 minutes, weigh about 2 g of the test sample into a previously ignited and weighed combustion boat. Weigh the absorption bulb. Return the absorption (see 6.2) bulb to the absorption assembly. Place the combustion boat containing the sample into the hot zone (900 to 915°C) of the furnace. Pass oxygen through the train for 30 minutes. Remove the absorption bulb from the train. Weigh the absorption bulb and combustion boat with material after combustion separately. The increase in mass in the bulb is due to absorption of carbon dioxide.

C-2.3 Calculation

Free carbon, percent by mass = $\frac{27.29 \ M_3 - (37.52) \ G}{M_1}$

where

 $M_1 = \text{mass in g of sample before combustion,}$

 M_2 = mass in g of sample after combustion,

 M_3 = mass in g of carbon dioxide absorbed (difference of initial and final mass of the absorption bulb), and

$$G = M_2 - M_1 + (.0.2729) M_3.$$

^{*}Specification for high temperature combustion boats.

C-3. DETERMINATION OF FREE SILICON DIOXIDE

C-3.1 Apparatus

C-3.1.1 Platinum Dish

C-3.1.2 Electric Muffle Furnace

C-3.1.3 Desiccator

C-3.2 Reagents

C-3.2.1 Hydrofluoric Acid — 48 percent (v/v).

C-3.2.2 Concentrated Nitric Acid — See IS: 264-1976*.

C-3.2.3 Concentrated Sulphuric Acid — See IS: 266-1977.

C-3.3 Procedure

C-3.3.1 Weigh about 3 g of dry test sample (see 6.2) into a weighed platinum dish which has been previously ignited to 700-800°C. Record the mass of the sample and the dish. Place the dish in an electric muffle furnace at 750 to 800°C for 20 minutes. Cool the dish in a desiccator.

C-3.3.2 Add 20 ml of hydrofluoric acid and 1 ml of nitric acid followed by 1-2 drops of sulphuric acid to the dish containing the sample.

C-3.3.3 Evaporate the solution to dryness on a sand bath. When the evolution of sulphur dioxide fumes ceases, ignite the dish along with the sample in the electric furnace. Cool the dish in a desiccator. Weigh the dish with content. Repeat the heating and cooling till a constant mass is obtained. The loss in mass is free silicon, free carbon and silicon dioxide. Retain the residue for determination of silicon carbide.

C-3.4 Calculation

Free silicon dioxide (SiO₂),
$$=\frac{(A \times 100) - (B + C)}{M}$$

where

A = mass loss in g (C-3.3.3),

B = percent by mass of free silicon determined in C-1.3,

C =percent by mass of free carbon determined in **C-3.3**, and

M = mass in g of sample.

^{*}Specification for nitric acid (second revision).

⁺Specification for sulphuric acid (second revision).

C-4. DETERMINATION OF SILICON CARBIDE

C-4.1 Reagents

- **C-4.1.1** Potassium Pyrosulphate ($K_2S_2O_7$).
- **C-4.1.2** Dilute Hydrochloric Acid (1:9) (v/v) See IS: 266-1977*.
- C-4.1.3 Concentrated Nitric Acid See IS: 264-1976.

C-4.2 Procedure

- **C-4.2.1** Add 10-15 g of potassium pyrosulphate ($K_2S_2O_7$) to the residue in the dish retained after determination of silicon dioxide (**C-3.3.2**). Fuse the content of the dish over a burner. Keep the fusion at dull red heat for 10 minutes. Cool the fusion and dissolve in 100 ml diluted hydrochloric acid in a 250 ml beaker. Remove the dish from the solution after completely washing with dilute hydrochloric acid.
- C-4.2.2 Filter the solution through filter paper. Wash the filter paper with dilute hydrochloric acid. Retain the filtrate for determination of aluminium oxide. Ignite the residue along with filter paper in the dish at 750-800°C to constant mass in the furnace. Record the mass of the dish and the silicon carbide.

C-4.3 Calculation

Silicon carbide (SiC), percent by mass
$$=\frac{100 \times M_1}{M}$$

where

 $M = \text{mass in g of sample (} \mathbf{C-3.3} \mathbf{)},$ and

 $M_1 = \text{mass in g of silicon carbide.}$

C-5. DETERMINATION OF SURFACE IRON OXIDE (Fe_2O_3)

C-5.1 Apparatus

C-5.1.1 Spectrophotometer

C-5.2 Reagents

- **C-5.2.1** Dilute Hydrochloric Acid (1:1)(v|v). See IS: 266-1977*.
- C-5.2.2 Sodium Acetate Solution 2 M. Dissolve 272 g of sodium acetate solution in 100 ml of water.

C-5.2.3 Bromophenol Blue Indicator

^{*}Specification for hydrochloric acid (second revision).

[†]Specification for nitric acid (second revision).

- C-5.2.4 Hydroxylamine Hydrochloride Dissolve 50 g of hydroxylamine hydrochloride in 500 ml of water. Filter, if necessary.
- **C-5.2.5** Orthophenanthroline Solution Dissolve 1 g orthophenanthroline monohydrate in 15 ml of ethanol [95 percent (v/v)]. Dilute to 1 000 ml with water.
- **C-5.2.6** Standard Iron Solution (1 ml = 0.02 mg of Fe). Dissolve 0.140.5 g of ferrous ammonium sulphate [Fe $(NH_4)_2$ $(SO_4)_2$, $6H_2O$] in 100 ml of dilute sulphuric acid (1:19) and dilute to 1000 ml in a volumetric flask.

C-5.3 Procedure

- C-5.3.1 Weigh about 10 g of the test sample (see 6.2) into a 400-ml beaker and add 40-50 ml of dilute hydrochloric acid. Cover with a watch glass and digest on a hot plate for about one hour. Filter through a filter paper into a 250 ml volumetric flask. Wash the residue with hot hydrochloric acid three or four times. Cool the volumetric flask to room temperature and dilute to mark.
- **C-5.3.2** Determine the volume of sodium acetate solution required for 5 ml of the solution from the volumetric flask (**C-5.3.1**) to attain the pH of 3.5 ± 1.0 using bromophenol blue as indicator. Transfer 5 ml of the solution into a 100 ml volumetric flask and add the same volume of sodium acetate solution.
- C-5.3.3 Add 10 ml of hydroxylamine hydrochloride (C-5.2.4) and 5 ml of orthophenanthroline solution (C-5.2.5) in water and make it up to the mark.
- C-5.3.4 Measure the optical density using a spectrophotometer at 510 nm against a blank in cell.
- C-5.3.5 Obtain the percent by mass of iron (Fe) using the standard calibration curve.

C-5.4 Calibration Curve

C-5.4.1 Take a series of standard iron solutions and the reagent blank using the same quantity of reagents. Carry through all the stages as described under C-5.3.2 to C-5.3.4 and record the photometric readings of all the standard solutions along the blank. Draw a calibration curve plotting the photometric reading of the standard solutions against milligrams of iron per 100 ml of the solution.

C-5.5 Calculation

Iron oxide, percent by mass = $1.43 \times \frac{A}{B} \times \frac{1}{10}$

where

A =mass in mg of iron found in the aliquot used, and

B =mass in g of the sample represented by the aliquot taken.

C-6. DETERMINATION OF ALUMINIUM OXIDE (Al_2O_3)

C-6.1 Reagents

- **C-6.1.1** Concentrated Buffer Solution of pH 6·1 Dissolve 27·5 g ammonium acetate and 11 g hydrated sodium acetate in 100 ml water. Add 1 ml glacial acetic acid and mix well.
- **C-6.1.2** Dilute Buffer Solution Add five volumes of water to one volume of concentrated buffer solution (**C-6.1.1**).
- C-6.1.3 Solochrome Cyanine-R Indicator Dissolve 0.1 g of solochrome cyanine-R in 100 ml water. The solution may be freshly prepared before use.
- **C-6.1.4** Standard Aluminium Solution (1 ml = 0.1 mg of aluminium). Dissolve 1.758 9 g of aluminium potassium sulphate [$Al_2(SO_4)_3K_2SO_4$. 24H₂O] in water, transfer to a 1 000-ml volumetric flask and dilute to the mark.
- **C-6.1.5** Standard Aluminium Solution (1 ml = 0.005 mg of aluminium) Transfer 25 ml of standard aluminium solution (**C-6.1.4**) to a 500 ml volumetric flask and dilute up to mark.

C-6.2 Apparatus

C-6.2.1 Spectrophotometer

C-6.3 Procedure

- C-6.3.1 Take the filtrate preserved in determination of silicon carbide (C-4.2.1) into a 250 ml volumetric flask and make it up to the mark.
- C-6.3.2 Transfer a suitable aliquot in a 100 ml volumetric flask, add 50 ml dilute buffer solution, adjust the pH to 6 and add 5 ml of solochrome cyanide-R indicator and make the volume to the mark and allow to stand for 5 to 10 minutes.
- C-6.3.3 Measure the optical density using a spectrophotometer at 535 nm against a blank solution in a cell.

C-6.3.4 Obtain the percent by mass of aluminium (Al) using a standard calibration curve.

C-6.4 Calibration Curve — Take a series of standard aluminium solutions and the reagent blank using the same quantity of reagents. Carry through all the stages as described under 6.3.1 and 6.3.3 and record the photometric readings of all the standard solutions along with blank. Draw a calibration curve plotting the photometric reading of the standard solutions against milligram of aluminium per 100 ml of the solution.

C-6.5 Calculations

Aluminium oxide, percent by mass = $1.89 \times \frac{A}{B} \times \frac{1}{10}$

where

A =mass in mg of aluminium found in the aliquot used, and

B = mass in g of the sample represented by the aliquot taken.

APPENDIX D

(Clause 6.1)

SAMPLING OF SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

D-1. SCALE OF SAMPLING

- D-1.1 All the containers in a consignment and belonging to the same variety and same batch of manufacture shall constitute a lot. If the consignment is known to consist of different batches of manufacture, the containers belonging to same batch and variety shall be grouped together and each such group shall constitute a separate lot.
- **D-1.2** Unless otherwise agreed to between the buyer and seller the number of containers to be selected shall depend upon the size of the lot and shall be in accordance with Table 4.
- **D-1.3** The sample shall be selected from the lot at random and in order to ensure randomness of selection, the method given in IS: 4905-1968* shall be followed.

^{*}Methods for random sampling.

TABLE 4 NUMBER OF CONTAINERS TO BE SELECTED FROM A LOT (Clause D-1.2)

Lot Size	Sample Size				
(1)	(2)				
Up to 25	3				
26 to 100	4				
101 to 150	5				
151 and above	7				

D-2. NUMBER OF TESTS

D-2.1 Tests for loose packed density and grain size shall be conducted on individual samples and for the remaining characteristics in Tables 2 and 3 shall be conducted on composite sample.

D-3. CRITERIA FOR CONFORMITY

D-3.1 For all those characteristics for which individual tests have been conducted average (\bar{X}) and range (\bar{R}) shall be calculated, range being the difference between the maximum and minimum of the test results and

Average
$$=\frac{\text{Sum of the test results}}{\text{Number of tests}}$$

D-3.2 The lot shall be declared as conforming to the specification with respect to grain size and loose packed density if:

$$R|UL - R|U - L \le 0.9;$$

 $\bar{X} + 0.6 R \le U \text{ and }$
 $\bar{X} - 0.6 R \ge L$

where

U is upper specification limit and L is the lower specification limit given in Table 2 and grain size if

 $\bar{X} - 0.6 R$ is equal to the minimum value specified.

D-3.3 For composite sample the lot shall be declared as conforming to the specification if it satisfied the relevant requirements in Tables 1, 2 and 3.

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IS 11643: 1985 SPECIFICATION FOR SILICON CARBIDE FOR BONDED ABRASIVE PRODUCTS

(Page 8, clause 5.2.1) — Substitute the following for the existing clause:

5.2.1 BIS Certification Mark

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.'

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(Page 9, clause 7.2, line 2) — Substitute 'IS 1070: 1992*' for 'IS: 1070 - 1977*'.
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(Page 9, footnote marked *) — Substitute the following for the existing:

(Page 10, clause **B-1.1.3**) — Substitute 'IS 2619 : 1993*' for 'IS : 2619 - 1971*'.

(Page 10, footnote marked *) — Substitute the following for the existing:

(Page 13, clause C-1.1.4) — Substitute 'IS 266: 1993*' for 'IS: 266 - 1977*'.

(*Page* 13, *footnote marked* *) — Substitute the following for the existing:

(Page 13, clause C-1.1.5) — Substitute 'IS 264: 2005†' for 'IS: 264-1976†'.

(Page 18, footnote marked †) — Substitute the following for the existing:

^{&#}x27;*Reagent grade water - Specification (third revision).'

^{&#}x27;*Glass beakers — Specification (second revision).'

[&]quot;*Sulphuric acid — Specification (third revision)."

^{&#}x27;†Nitric acid — Specification (third revision).'

Amend No. 1 to IS 11643: 1985

(Page 15, clause C-3.2.2) — Substitute *IS 264 : 2005** for *IS : 264 - 1976**.

(Page 15, footnote marked *) — Substitute the following for the existing:

**Nitric acid — Specification (third revision)."

(Page 15, clause C-3.2.3) — Substitute 'IS 266: 1993†' for 'IS: 266-1977†'.

(*Page* 15, *footnote marked* †) — Substitute the following for the existing:

"Sulphuric acid -- Specification (third revision)."

(Page 16, clause C-4.1.2) — Substitute 'IS 265: 1993*' for 'IS: 266 - 1977*'.

(Page 16, footnote marked *) — Substitute the following for the existing:

"Hydrochloric acid — Specification (fourth revision)."

(Page 16, clause C-4.13, line 1) — Substitute 'IS 264: 2005†' for 'IS: 264-1976†'.

(Page 16, footnote marked †) — Substitute the following for the existing:

'†Nitric acid — Specification (third revision).'

(CHD 9)